

Synthesis and Characterization of New Complexes of (N-4-Methoxy Phenyl) Amino Phenyl Acetonitrile Ligand with Some Divalent Transition Metal Ions

Mohammed H. A. Al-Amery

Department of Chemistry, College of Science, Baghdad University.

Abstract

The synthesis and characterization of new complexes of Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) with bidentate (N-4-methoxy phenyl) amino phenyl acetonitrile ligand which was prepared from benzaldehyde and 4-methoxy aniline in the presence of KCN and acidic medium have been described. The complexes were synthesized by treating an ethanolic solution of the ligand with appropriate amount of metal salts [1:2] [M:L] ratio. The complexes were characterized by metal and elemental analysis, infrared and electronic spectra, molar conductivity, magnetic susceptibility, and mole ratio method. According to obtained data the probable coordination geometries of Manganese, Cobalt, Nickel, Copper, and Zinc in these complexes with N₂-donor of ligand atoms are octahedral. All complexes were found to be non-electrolyte in absolute ethanol, and the complexes were formulated as [ML₂Cl₂].XH₂O.

Keywords: α -aminonitrile, Manganese, Cobalt, Nickel, Copper and Zinc Complexes.

Introduction

The classical Strecker reaction provides one of the most efficient methods for the synthesis of α -aminonitriles since 1850 when Adolph Strecker interact an aldehyde, an amine and hydrogen cyanide. The addition of cyanide to imines provides a direct route for the synthesis of α -aminonitriles. Some of the α -aminonitrile derivatives like 1-amino-4-phenylnaphthalene-2-carbonitrile have been postulated to have high fungistatic activity even stronger than the activity of the commercial fungicide-Kaptan [1]. α -aminonitriles are useful intermediates for the synthesis of amino acids [2,3] and Nitrogen-containing heterocycles such as thienopyrimidine derivatives which have antibacterial and antifungal activities higher than the corresponding antibacterial ampicillin and antifungal nystatin [4-6]. Moreover, among many other applications, they are readily hydrolyzed to diamines, which are of interest as ligands for Platinum (II) complexes with potential antitumor properties [7,8]. Several modifications of the Strecker reaction have been reported using a variety of cyanating agent such as α -trimethylsiloxynitriles and under various reaction conditions [9,10]. Complexes with dinitrogen N₂ ligand are of interest not only in theoretically but also from a practical point of view. They have found application as

antitumor activity [11], antibacterial activity [12] and antiviral activity in agriculture field [13]. α -aminonitriles have often been used as chelating ligands through two nitrogen atoms in the field of amino coordination chemistry. This is the first study to prepare and characterize the complexes of α -aminonitrile compounds with transition metal ions to reveal a new coordination field of these compounds as ligands with metal ions.

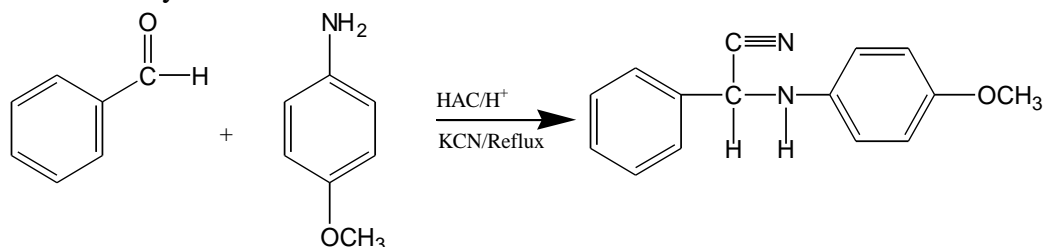
Experimental

All the chemicals and solvents used for the synthesis were of reagent grade and were obtained commercially from Fluka company with the exception of MnCl₂.4H₂O, CoCl₂.6H₂O, NiCl₂. 6H₂O, CuCl₂.2H₂O and ZnCl₂ salts were obtained from British Drug House (BDH). The prepared ligand (N-4-methoxy phenyl) amino phenyl acetonitrile was synthesized and characterized according to published work. The infrared spectra of the ligand and the complexes were recorded on a Shimadzu (8400s) FTIR Spectrophotometer, as CsI disk. Electronic absorption spectra were recorded in the range (200 – 1100) nm on a Shimadzu (160 A) Spectrometer in freshly prepared 10⁻³ M in absolute ethanol at room temperature using quartz cell (1.00) cm. Atomic absorption technique was used to determine the metal contents of the complexes using a Shimadzu (A.A 680G) Atomic absorption

spectrophotometer. Molar conductivity was used to measure the conductivity of the complexes at room temperature in freshly prepared 10^{-3} M in absolute ethanol using (PW9526) Digital conductivity meter. Elemental analysis for Carbon, Hydrogen and Nitrogen elements were carried out at the micro analytical Center/ Cairo University, Giza, Egypt. Gallen Kamp apparatus was used to determine the melting points of the ligand and the prepared complexes. Also the chlorine content was determined gravimetrically [14].

Preparation method of (N-4-methoxy phenyl) amino phenyl acetonitrile ligand [L]

A general method [15, 16] can be adopted for [L] preparation which can be described as follows: The benzaldehyde (1 mmol) was added to (20 ml) glacial acetic acid, p-toluene sulphonic acid was added in very small portion as catalyst, followed by addition of (1 mmol) p-methoxy aniline. The pH was adjusted to about 4 by addition of concentrated

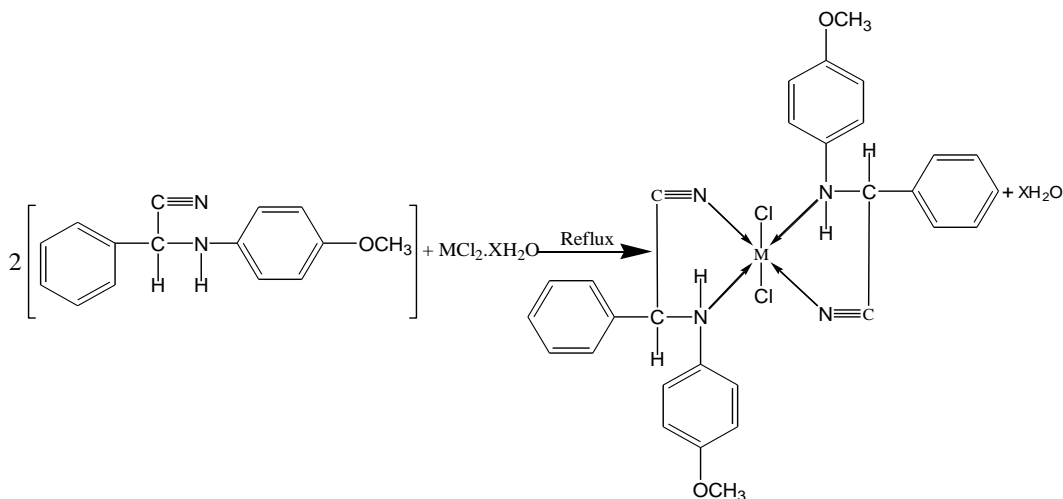


Preparation method of metal complexes

The complexes $[ML_2Cl_2].XH_2O$ have been prepared by the reaction of (0.476 gm) (2 mmol) of (L) with (1mmol) of metal chloride (0.198 gm, 0.238 gm, 0.238 gm, 0.171 gm, and 0.136 gm for $MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and $ZnCl_2$ respectively) dissolved in (20 ml)

sulfuric acid drop wise to obtain Schiff base which stirred for 30 min. Potassium cyanide (2 mmol) was added to the mixture and kept under stirring for 4 days. The reaction mixture was poured into ice and then made slightly alkaline with ammonium hydroxide solution. The solid precipitate which formed was filtered, washed with water and air dried. The presence of nitrile group in the prepared α -aminonitrile was indicated by treating few amount of the sample with 10% sodium hydroxide solution, the liberation of ammonia after hydrolysis of nitrile group, which was detected by wet red litmus paper, indicating the presence of nitrile group. Purity of the obtained compound was checked by TLC, using chloroform and ethyl acetate (1:1) as eluent. The product color was light green and the yield percentage was (85.23 %) and the melting point was (58-60 $^{\circ}C$). The reaction was clarified below:

absolute ethanol and refluxed with stirring under anhydrous conditions using Na_2SO_4 (anhydrous) for 24 hours. The obtained complexes were collected after evaporation of ethanol and triturated with petroleum ether (60-80 $^{\circ}C$) then filtered and the products were left in the desiccator to be dried under P_2O_5 . The general reaction was clarified below:



Results and Discussion

All the compounds reported in this work are presented in Table (1) which illustrated physical properties, along with their molar conductivity at room temperature. Metal and elemental analysis of all compounds are represented in Table (2). The ligand (N-4-methoxy phenyl) amino phenyl acetonitrile [L] was prepared as reported in literature [17]. It was characterized by elemental analysis and infrared spectral data. The complexes have been prepared under anhydrous conditions to avoid any hydrolysis of nitrile group to amide due to the presence of water molecules and metal ion [18]. In addition to that when organic molecules are coordinated to metal cations in organo-metallic or coordination compounds, they become susceptible to nucleophilic attack like water molecules which cause hydrolysis of nitrile into amide [19]. These complexes were stable in the solid state and were characterized by the usual methods; Molar conductivity, mole ratio, metal and elemental analysis, chloride content, magnetic susceptibility, FTIR and UV-Visible spectroscopy. On the basis of the characterization methods it's suggested that all complexes probably have octahedral structure with coordination number 6 that includes two atoms of coordinated chlorides. The conductivity values for the complexes of ($10^{-3}M$) in absolute ethanol were recorded in the range (4-12) indicating that the non-electrolytic nature of the complexes [20]. Mole ratio method was used to determine the ratio of metal ion to the ligand for the complexes at λ_{max} ($\lambda_{max} = 571nm$ for $[CoL_2Cl_2].5H_2O$ and $\lambda_{max} = 460 nm$ for $[CuL_2Cl_2].2H_2O$) in alcoholic solutions.

Infrared Spectroscopy

The infrared spectral data of [L] in Table (3) showed some characteristic bands related to α -aminonitriles; Like $2200-2300 cm^{-1}$ which could be attributed to $C\equiv N$ group [21]. A shift of the range $65-141 cm^{-1}$ was observed for $C\equiv N$ stretching vibration on coordination due to the decrease of bond order as result of metal nitrogen bond formation. The next characteristic band near $1600 cm^{-1}$ which could be attributed to N-H group. A red shift of the range $9-27 cm^{-1}$ was observed in N-H bending frequency on complexes

indicating the coordination on nitrogen to metal ion. On the other hand the spectrum of the complexes showed new bands around $580-600 cm^{-1}$ and $355-390 cm^{-1}$ due to $\nu M-N$ and $\nu M-Cl$ respectively [22, 23]. The presence of these bands supported the formation of complexes under investigations. The presence of lattice water molecule in all complexes Table (3) was supported by the broad band in $3500-3150 cm^{-1}$ region of infrared spectrum [22] and by its metal and elemental analysis data (calculated) in Table (2).

Ultra Violet – Visible Spectroscopy

The ultra violet spectral data of the ligand, the complexes and the magnetic moment values are listed in Table (4). The ligand showed two absorption bands in the region $32786 cm^{-1}$ and $29325 cm^{-1}$ corresponding to $(\pi \rightarrow \pi^*)$ and $(n \rightarrow \pi^*)$ transitions respectively. On complexation shifting has been observed in ligand field and new bands were observed due to the presence of metal ions in the complexes. The electronic spectrum of Zinc (II) complex showed only the charge transfer transition of $25316 cm^{-1}$ which can be assigned to charge transfer from the metal to the ligand ($M \rightarrow L$) no ($d \rightarrow d$) transition are expected for d^{10} Zinc (II) complex [24]. The electronic spectrum of Copper (II) complex showed two bands. These two bands appeared at $14534 cm^{-1}$ and $21739 cm^{-1}$ ascribed to the ($d \rightarrow d$) transitions. A band of $[CuL_2Cl_2].2H_2O$ complex spectrum appeared above $25974 cm^{-1}$ represented to the ($L \rightarrow M$) charge transition band and the obtained value of the effective magnetic moments (1.82BM) is typical for distorted octahedral Cu(II) chelates. The electronic spectrum of Nickel (II) complex showed two ($d-d$) transition bands at $16806 cm^{-1}$ and $19607 cm^{-1}$ corresponding to ${}^3A_{2g}(F)$ ground state, ${}^3T_{1g}(F)$ and ${}^3T_{1g}(P)$ in the excited state respectively which suggested mostly octahedral geometry. The magnetic moment value for the Nickel (II) complex of the ligand at $25^\circ C$ was found to be (2.72 B.M) [25-27]. A peak of $[NiL_2Cl_2].H_2O$ complex spectrum appeared at $25510 cm^{-1}$ which represented to the ($L \rightarrow M$) charge transition band. $[CoL_2Cl_2].5H_2O$ complex has magnetic moments value (3.78 B.M) which agreed with the expected value for the high spin Cobalt(II)

ion in octahedral environment. The electronic spectrum of Cobalt (II) complex showed two main bands at 17513 cm^{-1} and 19685 cm^{-1} . These two bands are assigned to ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ (ν_2) and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ (ν_3) transitions respectively. The (ν_1) transition is expected to appear at large wavelength. Therefore it could not be observed, it will be calculated theoretically through the Tanabe-Sugano diagram. Therefore it could not be observed, while the observed transitions are consistent with an

octahedral geometry. A band of Co(II) complex spectrum appeared at 24390 cm^{-1} represented to the (L \rightarrow M) charge transition band [28, 29]. The electronic spectrum of Manganese (II) complex showed only the charge transfer transition of 26666 cm^{-1} which can be assigned to charge transfer from the metal to the ligand and (M \rightarrow L) no (d \rightarrow d) transition are expected for d^5 (half-filled) for $[\text{MnL}_2\text{Cl}_2].5\text{H}_2\text{O}$ complex because its Term symbol is 6S [30,31]

Table (1)
Physical properties of (L) and its metal complexes with their conductivity values.

Compound	Color	M.P. °C	Yield %	$\Omega^{-1}\text{cm}^2\text{mol}^{-1}$
L	Light green	58-60	85.23	-----
$[\text{MnL}_2\text{Cl}_2].5\text{H}_2\text{O}$	Green yellow	187-189	82.17	9
$[\text{CoL}_2\text{Cl}_2].5\text{H}_2\text{O}$	Green	222-224	80.63	4
$[\text{NiL}_2\text{Cl}_2].\text{H}_2\text{O}$	Light blue	>330	75.85	12
$[\text{CuL}_2\text{Cl}_2].2\text{H}_2\text{O}$	Brown	108-110	86.12	11
$[\text{ZnL}_2\text{Cl}_2].\text{H}_2\text{O}$	Pale green	178-180	83.39	6

Table (2)
Analytical data of the (L) and its Complexes.

Formula M.wt. (gm/mole)	Elemental Analysis Calculated (Found)				
	C%	H%	N%	M%	Cl%
L 238	75.63 (75.02)	5.88 (5.38)	11.65 (11.59)
$[\text{MnL}_2\text{Cl}_2].5\text{H}_2\text{O}$ 691.94	52.03 (51.93)	5.49 (5.68)	8.09 (8.06)	7.94 (7.33)	10.25 (10.44)
$[\text{CoL}_2\text{Cl}_2].5\text{H}_2\text{O}$ 695.94	51.73 (51.87)	5.46 (5.61)	8.05 (8.38)	8.47 (8.84)	10.19 (10.39)
$[\text{NiL}_2\text{Cl}_2].\text{H}_2\text{O}$ 623.69	57.72 (57.95)	4.81 (4.64)	8.98 (8.72)	9.41 (9.01)	11.37 (11.42)
$[\text{CuL}_2\text{Cl}_2].2\text{H}_2\text{O}$ 646.55	55.68 (55.56)	5.26 (5.83)	8.66 (9.28)	9.83 (9.27)	10.97 (11.38)
$[\text{ZnL}_2\text{Cl}_2].\text{H}_2\text{O}$ 630.28	57.12 (58.17)	4.76 (4.71)	8.88 (8.67)	10.37 (10.52)	11.25 (11.13)

Table (3)
The characteristic bands of FTIR spectra of the ligand and its metal complexes in (cm^{-1}).

Compound	$\nu\text{O-H cm}^{-1}$ Lat. water	$\nu\text{C-H cm}^{-1}$ Aromatic	$\nu\text{C-H cm}^{-1}$ Aliphatic	$\nu\text{C}\equiv\text{N}$ Nitrile	$\delta\text{N-H}$	$\delta\text{C-H out of plane}$		$\nu\text{M-N}$	$\nu\text{M-Cl}$
						Mono subst.	Para subst.		
L	-----	3016	2970	2237	1627	702,756	817	-----	-----
[MnL ₂ Cl ₂].5H ₂ O	3379	3024	2955	2160	1600	705,725	833	595	380
[CoL ₂ Cl ₂].5H ₂ O	3417	3070	2931	2168	1612	704,755	825	600	373
[NiL ₂ Cl ₂].H ₂ O	3413	3051	2950	2172	1618	700,752	802	580	390
[CuL ₂ Cl ₂].2H ₂ O	3337	3062	2939	2145	1612	702,764	833	593	355
[ZnL ₂ Cl ₂].H ₂ O	3450	3042	2926	2096	1618	671,733	825	585	365

Table (4)
Electronic spectra in absolute ethanol for the (L) and its metal complexes.

Compound	λ nm	ν cm^{-1}	Transition	B^- cm^{-1}	Dq/B^-	$10Dq$ cm^{-1}	β	$15B^-$ cm^{-1}	Suggested structure
L	305 341	32786 29325	$\pi \rightarrow^* \pi$ $n \rightarrow^* \pi$						
[MnL ₂ Cl ₂].5H ₂ O	282 350 375	35461 28571 26666	Ligand Field Ligand Field C.T						O.h
[CoL ₂ Cl ₂].5H ₂ O	337 351 410 508 571	29673 28490 24390 19685 17513 8859(cal.)	Ligand Field Ligand Field C.T ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$ ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$ ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$	708	1.222	8637.6	0.729	10620	O.h
[NiL ₂ Cl ₂].H ₂ O	318 355 392 510 595	31446 28169 25510 19607 16806 8005(cal.)	Ligand Field Ligand Field C.T ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$	827	1.064	8799.2	0.803	12405	O.h
[CuL ₂ Cl ₂].2H ₂ O	325 352 385 460 688	30769 28409 25974 21739 14534	Ligand Field Ligand Field C.T ${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$						distorted O.h
[ZnL ₂ Cl ₂].H ₂ O	290 335 395	34482 29850 25316	Ligand Field Ligand Field C.T						O.h

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وفي وسط حامضي. تم تخليق المعقدات بمعاملة محلول كحولي لليكاند مع كمية مناسبة من املاح الفلزات ونسبة [M:L] [1:2]. تم تشخيص المعقدات المحضرة بواسطة تحليل العناصر والمعادن وطيف الأشعة تحت الحمراء وطيف الاشعة فوق البنفسجية المرئية والتوصيلية المولارية والحساسية المغناطيسية وطريقة النسبة المولية. طبقاً إلى البيانات المستحصلة فالأشكال الفراغية المحتملة للتناسق لكل من معقدات المنغنيز والكوبلت والنيكل والنحاس والخراسين مع ذرات النايتروجين المانحة لليكاند هي ثمانية السطوح. لقد تبين ان كل المعقدات هي غير الكتروليتية في مذيب الإيثانول المطلق وهذه المعقدات لها الصيغة العامة $[ML_2Cl_2].XH_2O$.

الخلاصة

تم وصف تخليق وتشخيص معقدات جديدة لبعض الايونات الفلزية لكل من Ni(II), Co(II), Mn(II) Zn(II), مع الليكاند (4-N-ميثوكسي فنيل) امينوفنيل اسيتونتريل ثنائي السن والذي تم تحضيره من مفاعلة البنزالديهيد مع باراميثوكسي انيلين بوجود سيانيد البوتاسيوم